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# THE EFFECTS OF HARD ANODIZING ON THE STRESS CORROSION CRACKING OF ALUMINUM ALLOYS

by

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ABSTRACT

The breaking load method was used to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens of AA 7075-T651 in order to evaluate the benefits of anodizing the aluminum pressure hull of an autonomous underwater vehicle to reduce the risk of stress corrosion cracking. The original test method showed that anodizing delayed the onset of stress corrosion cracking of specimens subjected to an externally-applied stress of 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an external stress. The value of the threshold stress determined for the uncoated specimens was within the range reported by an interlaboratory test program.

The extreme value distribution was found to provide a good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory program. A new statistical procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusion reached using the original method, even though the statistical calculations may not have been strictly valid. The new procedure also showed that hard anodizing continued to offer improved resistance to stress corrosion cracking at higher stress levels. Finally, analysis of variance of the transformed data confirmed the conclusion was that anodizing provided a significant improvement in resistance to stress corrosion cracking, with hard anodizing providing the greater benefit.

## 1. INTRODUCTION

An autonomous underwater vehicle being designed and built for Canada's Department of National Defence will be subjected to repeated exposure to sea water during its testing and deployment. Its pressure hull will be constructed using a number of cylinders made from Aluminum Alloy (AA) 7075-T7351, an alloy that was originally developed for high strength applications in aircraft and which normally requires protection in severely corrosive environments such as sea water[1]. The T7351 temper was developed to improve the resistance of this alloy to stress corrosion cracking (SCC) but cracking has still been observed in the laboratory environment[2]. The original design did not call for any corrosion protection for the pressure hull but concerns about SCC led to the specification of a hard anodized coating for the aluminum alloy.

Some early research suggested that anodizing accelerated the SCC of aluminum alloys[3-5] but Skoulikidis and co-workers[6-9] demonstrated that a properly applied, sealed coating had a protective effect by increasing the time to failure of specimens loaded in tension. A new procedure for determining the susceptibility of aluminum alloys to SCC that does not depend on traditional time to failure methods has been developed by Alcoa Laboratories for the National Aeronautics and Space Administration. This procedure, known as the breaking load method[10], appeared to have the ability to separate the effects of SCC from the damage caused by pitting and general corrosion. However, a recent interlaboratory test program aimed at assessing the reproducibility of the breaking load method revealed several shortcomings in the statistical procedure that was used[11]. An alternate approach, based on Box-Cox transformations[12], was proposed and tested.

In order to evaluate the effectiveness of anodizing the pressure hull to reduce the risk of SCC, the Defence Research Establishment Pacific (DREP) has used both the original breaking load method and the proposed modification incorporating the Box-Cox transformation to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens made from AA 7075-T651. The susceptibility of this temper to SCC has been extensively studied by traditional methods, to the point that it appears as an example in ASTM G47-79[13] and was used in the interlaboratory test program[11]. It was chosen for this study because tensile specimens were already available and there were severe time constraints associated with the decision on whether to specify an anodized coating for the hull components. Since this temper is more

susceptible to SCC than the T7351, it also served to highlight any improvements that anodizing might produce.

## 2. BACKGROUND ON THE BREAKING LOAD METHOD

The raw data for the breaking load method is obtained by measuring the "apparent tensile strength" of smooth tension specimens that have been stressed to pre-determined levels in a "constant strain" fixture and then exposed to the corrosive environment for set periods of time. The apparent tensile strength of each specimen is calculated using its original cross-sectional area without consideration for any thinning of the specimen due to general corrosion or stress corrosion cracking. This data provides an indication of the change in load carrying capability of the specimens as a function of exposure time and externally-applied stress. Reductions in the apparent tensile strength for stressed specimens beyond those found for unstressed specimens are assumed to be due to stress corrosion cracking.

Statistical analysis of the breaking load data is based on the assumption that the specimen breaks at its weakest point. If a specimen develops SCC, it may contain a wide spectrum of crack sizes but fracture will always occur at the largest crack. As a result, the distribution of all crack sizes within a group of specimens is not as important as the distribution of the largest cracks. The extreme value distribution pioneered by Gumbel[14] applies to the largest cracks, assuming that the distribution of all crack sizes is exponential. These assumptions were supported by the work of Aziz[15], who characterized pitting depth with an exponential distribution and showed that the maximum pit depth followed the related extreme value distribution. Since SCC is also related to the microstructure, the breaking load data for specimens suffering from SCC should follow an extreme value distribution as well. However, since fracture stress is related to the largest flaw size in an inverse manner, the appropriate distribution for a fracture stress would be an extreme value distribution of smallest values[10]. This distribution is bell-shaped but skewed to the left, so a group of replicate tests is likely to produce a wider range of fracture stresses below the most probable value than above.

The probability for survival, with survival defined as no failure at the stress of interest during the period of exposure, is expressed by the following equation, attributed to Gompertz[16]:

$$P = \exp(-e^{z}) \qquad (1)$$

where P is the probability of survival and Z is the reduced variate, which has the form:

$$Z = (S-\beta)/\theta \qquad (2)$$

where S is the exposure stress and $\beta$ and $\theta$ are estimates of the distribution location and scale parameters, respectively. These parameters, which are comparable to the mean and standard deviation of the normal distribution, are determined using the probability plotting method[17]. The breaking strengths of the replicate specimens in a test group are ranked in descending order and plotted against the expected value of each ranked observation. Specimens which broke prior to tensile testing are assumed to belong to the distribution, and hence affect the expected values, but are not available for plotting.

The expected value of the $i^{th}$ observation is estimated by the $R^{th}$ fractile of the distribution, where:

$$R_i = (i-\frac{1}{2})/N \qquad (3)$$

and N is the sample size. For the extreme value distribution of smallest values, the expected value of the $i^{th}$ observation is:

$$E_i = -\ln[-\ln(R_i)] \qquad (4)$$

If the data from a test group follows an extreme value distribution, a plot of $E_i$ versus the ranked breaking strengths will yield a straight line. The slope, m, and intercept, b, of this line are determined by linear regression and provide estimates of $\beta$ and $\theta$ for a large sample size, denoted by $\beta_\infty$ and $\theta_\infty$, from the following equations:

$$\beta_\infty = -\frac{b}{m} \qquad (5)$$

$$\theta_\infty = -\frac{1}{m} \qquad (6)$$

Before their use in Equation 2, these parameters must be adjusted to account for the small sample size employed in our experiments according to the following equations:

$$\theta = \theta_\infty(\sigma_\infty/\sigma_n) \qquad (7)$$

$$\beta = \beta_\infty - \theta_\infty y_\infty + \theta \bar{y}_n \qquad (8)$$

where n is the number of the N specimens which survived to be tensile tested, $\sigma_\infty = \pi/\sqrt{6}$, $y_\infty = 0.577$, and $\sigma_n$ and $\bar{y}_n$ are found in a table extrapolated from the work of Gumbel[18] by Sprowls and co-workers[19].

One of the advantages reported for the breaking load method is the ability to estimate the survival probability P (Equation 1) with reasonable confidence as the actual probability of survival for a resistant material approaches 1.0. Such a material normally displays very few failures in traditional time-to-failure methods. As a result, a prohibitively large number of specimens would be required to determine a value of P with reasonable confidence.

A more meaningful use of breaking load data can be obtained by calculating the tensile stress at which 99% of the specimens could be expected to survive a specified exposure stress for a specified length of time. This 99% survival stress ($S_{99}$) is determined using the following equation:

$$\begin{aligned} S_{99} &= \beta + \theta \cdot \ln[\ln(0.99)^{-1}] \\ &= \beta - 4.60 \cdot \theta \end{aligned} \qquad (9)$$

and not only permits a direct comparison of stressed and unstressed specimen performances, but also provides a numerical value for comparing materials. Furthermore, the 99% survival stress can be used with fracture mechanics theory to estimate the equivalent flaw size that would not be exceeded in 99% of the SCC specimens that survived. A plot of this flaw size as a function of time could provide information about crack growth rates.

The 99% survival stress obtained for a given material at a specified exposure stress and length of exposure does not generally correspond to a threshold stress below which SCC would not be expected to occur within the specified exposure time. However, a series of $S_{99}$ values can be used to obtain a statistically-defined threshold stress, $S_{th}$, such as the stress for which there is a 95% confidence of a probability of survival greater than 99%. This threshold stress is determined by plotting $S_{99}$ against the exposure stress for each exposure time and calculating the intersection of each best fit line, obtained by linear regression analysis, with the line defined by $S_{99}$ equalling the exposure stress. The mean, m, and standard deviation, s, of these

intersection points are calculated and then used to determine the lower confidence limit, LCL, from the following equation:

$$LCL = S_{th} = m - \frac{t_{n-1} \cdot s}{\sqrt{n}} \qquad (10)$$

where n is the number of different exposure times (intersection points) and the value of $t_{n-1}$ is obtained from a table of the cumulative t distribution[20] for a one-tailed t-test with 95% confidence. The value of $S_{th}$ provides a single number which can be used to rate the behaviour of different alloys in the same environment or, in our case, the same alloy with different surface treatments.

## 3. BOX-COX TRANSFORMATIONS

A major problem with the original breaking load method that was identified by the interlaboratory test program is the extreme sensitivity of the value of the 99% survival stress to differences in the standard deviation of different test groups. Colvin and Emptage[11] showed an example of data from an identical test group (five specimens of AA 7075-T651 exposed to alternating immersion in 3.5% sodium chloride for 6 days at 138 MPa) from two different laboratories. From the number of specimens which failed prior to tensile testing, it was clear that one group had suffered more damage than the other. However, the calculated value of $S_{99}$ was lower for the group with more survivors, simply because there was a greater variation between individual breaking strengths within that group. Furthermore, eliminating the worst performing specimens (those that broke prior to tensile testing) from the calculations of $\mu$ and $\sigma$ biased the test results, leading to higher values of $S_{99}$ for groups in which some specimens failed than would occur if those specimens had barely survived to be tested. Use of the extreme value distribution could also give rise to negative values for $S_{99}$, and hence for $S_{th}$, which are physically impossible. The obvious first approach of setting negative values of $S_{99}$ to zero was shown to result in misleading conclusions. Finally, the statistical procedure could only be applied to the individual stress/time test groups. As a result, the method could not be used to obtain an overall estimate of the variance which would account for inconsistencies not related to resistance to SCC, such as machining finish and location within the exposure apparatus.

The Box-Cox transformation was proposed to address the problems encountered with the extreme value distribution. Successful implementation of the transformation is predicated on the assumption

that the variance increases in some manner with decreasing residual strength. This assumption seems reasonable for specimens which are experiencing stress corrosion cracking. Prior to the initiation of SCC, there should only be a small variation in the breaking strengths of individual specimens. However, as cracks grow, and the breaking strength decreases, there should be an ever increasing variation in crack lengths resulting from even slight differences in crack growth rates between specimens.

The first step in the procedure involves a preliminary transformation of the original breaking strength values, X, by the following equation:

$$X_{tr} = \frac{100 \cdot X}{X_0} \qquad (11)$$

where $X_0$ is the average breaking load for no exposure for the given alloy or temper. The value of $X_{tr}$ provides the percent retention of the original breaking strength and therefore standardizes the results for alloys with different uncorroded strength levels.

The average, m, and the standard deviation, s, are calculated for each individual stress/time test group with more than one specimen which survived to be tensile tested. The slope, $\alpha$, of the best fit straight line is determined from the plot of ln(s) versus ln(m) for each different alloy or, in our case, different corrosive environment. This plot describes the nature of the relationship assumed to exist between the breaking strength and the variance. If the plot is not linear, the Box-Cox procedure cannot be applied to the system under investigation. If a meaningful value of $\alpha$ can be determined, the Box-Cox transformation is a power transformation which has the form:

$$Y = (100/100^{\lambda}) \cdot (X_{tr}^{\lambda} - 1) \qquad (12)$$

where the power of transformation, $\lambda$, is equal to $1 - \alpha$. The constant, $100/(100)^{\lambda}$, gives rise to transformed values of Y in the range from 0 to 100, which is the same range as the values of $X_{tr}$.

As shown in Figure 1, the Box-Cox transformation essentially stretches the residual strength axis for large strength values and compresses the axis for small values. As a result, the transformation provides a means of discriminating between alloys with high levels of resistance to SCC. Such alloys would be difficult to separate using traditional pass/fail criteria. Another important effect of the

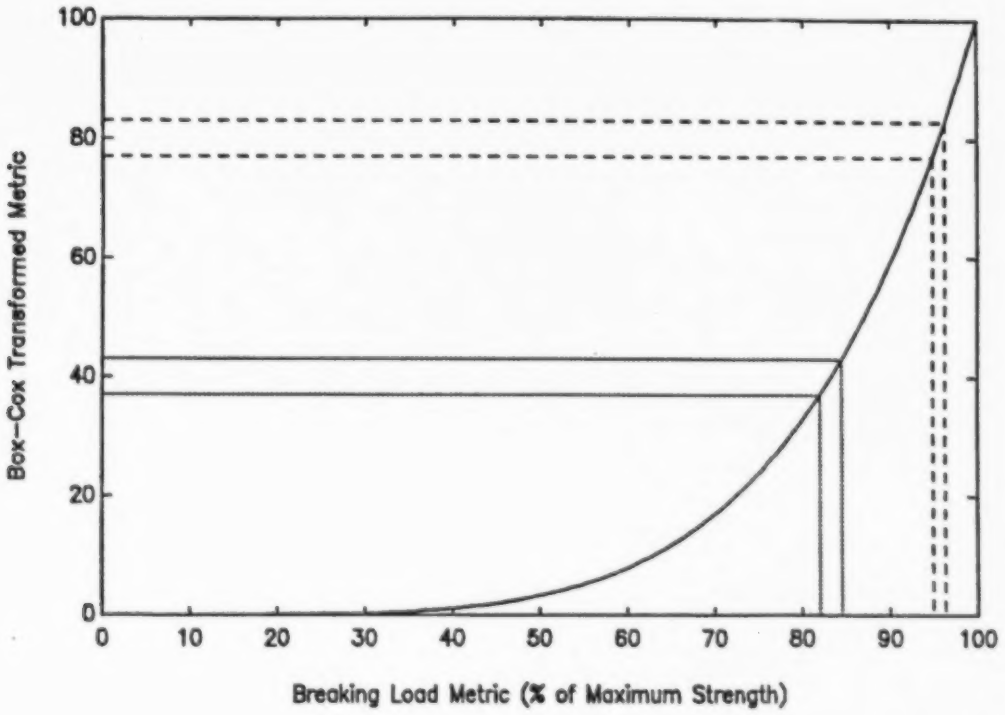


Figure 1. Box-Cox transformation curve for the example of $\lambda$=5. The dashed lines illustrate the transformation of the variance for one hypothetical test group while the dotted lines illustrate that for another test group with lower breaking strength and larger variance.

transformation is also illustrated in Figure 1. The variances of the individual stress/time test groups, which increase with decreasing mean breaking strength, are all approximately equal after transformation. Furthermore, the transformed data follows a normal distribution in many cases. As a result, standard statistical techniques, such as t-tests and ordinary analysis of variance[21] (ANOVA), can be used. Since the variance is equivalent throughout the transformed Box-Cox metric, least significant differences and lower confidence limits are easily calculated.

Once the value of $\lambda$ has been determined, statistically plausible values of Y are generated for the specimens which fail prior to tensile testing. A specimen which fails on the day it is scheduled to be tensile tested is assigned a value of Y equal to the transform of the exposure stress, $Y_{exp}$. A specimen which fails prior to the final day is assigned a value of Y obtained by generating a random number over the

interval $(0, Y_{exp})$. This procedure is simple, conservative and allows the use of the analysis of variance technique.

A simple way of analyzing a set of transformed data would involve the determination of the mean and standard deviation of each cell (stress/time test group) in the data table. Since each cell has the same number of "observations", the pooled estimate of the standard deviation, $s_p$ is:

$$s_p = \sqrt{\frac{(s_1^2+s_2^2+\ldots+s_r^2)\,(N-r)}{r\,(N-r-c)}} \tag{13}$$

where N is the total number of observations (specimens), r is the number of data cells and c is the number of specimens that failed prematurely. The smallest difference in the means of two cells that is statistically significant, the least significant difference or LSD, is given by:

$$LSD = t_\upsilon s_p \sqrt{\frac{2}{n}} \tag{14}$$

where n is the number of observations per cell, the degrees of freedom, $\upsilon$, are given by:

$$\upsilon = N-r-c, \tag{15}$$

and $t_\upsilon$ is obtained from a table of the cumulative t distribution[20] for a two-tailed t-test with 95% confidence. The value of LSD can be used to compare two cells to determine whether or not the data in them comes from two populations with different means.

In order to compare cells from two different alloys or corrosive environments, the estimated variances from the two data sets must first be pooled. For data sets 1 and 2, with variance estimates, $s_1^2$ and $s_2^2$, and degrees of freedom, $\upsilon_1$ and $\upsilon_2$, respectively, the pooled standard deviation is given by:

$$s_p = \sqrt{\frac{\upsilon_1 s_1^2+\upsilon_2 s_2^2}{\upsilon_1+\upsilon_2}} \tag{16}$$

A lower confidence limit for the mean value of any data cell can be calculated from the expression:

$$LCL = m_{BC} - \frac{t_v s_p}{\sqrt{n}} \qquad (17)$$

where $m_{BC}$ is the average Box-Cox transformed value and $t_v$ is obtained from a table of the cumulative t distribution[20] for a one-tailed t-test. The LCL values can then be transformed back to either the $X_{tr}$ or the original X metrics.

## 4. EXPERIMENTAL PROCEDURE

Round tension specimens with a diameter of 3.18 mm, a gauge length of 25.4 mm and an overall length of 50.8 mm were machined from a 76.2 mm thick plate of AA 7075-T651 in the short transverse orientation in accordance with ASTM Standard G49-76[22]. Chemical analysis of this plate is shown in Table 1. One group of eighty-five specimens was hard anodized under industrial conditions by the Ship Repair Unit (Pacific) according to U.S. Military Specification MIL-A-8625F Type III. A second group received a sulphuric acid anodized coating according to MIL-A-8625F Type II. A third group was left as-machined.

Tensile stresses of 0, 69, 138 or 207 MPa were applied to the specimens using a wedge-type, constant-strain frame as shown in Figure 2. The frames were assembled finger-tight and then placed in a commercially-available, synchronous loading device that was developed by Alcoa Research Laboratories[23]. Operation of the device caused the inward movement of the wedge-shaped side pieces of the stressing frame, producing an uniaxial tensile stress in the specimen. This stress was related directly by Hooke's law and Young's modulus to the strain of the specimen, which was measured with an MTS model 623.13B-20 extensometer that had a 12.7 mm gauge length. Friction between the various components of a stressing frame maintained a constant strain after removal of the frame from the loading device.

Sets of five replicate specimens of each of the four stress levels for each of the three surface treatments were exposed to

TABLE 1. Chemical Composition of AA 7075-T651 Specimens

| Element | Cu | Mg | Zn | Mn | Fe | Si | Cr | Ti | Al |
|---|---|---|---|---|---|---|---|---|---|
| Weight percent | 1.39 | 2.45 | 5.92 | 0.06 | 0.15 | 0.09 | 0.2 | 0.03 | rem |

alternate immersion in sea water in accordance with the procedure described in ASTM Standard G44-75[24] for 2, 4, 7 or 10 days. A total of eighty specimens were exposed for each surface treatment. The specimens were continuously immersed in the sea water for 10 min of each hour and then allowed to dry for the remaining 50 min. This cycle was achieved by placing a pump on a timer. While the pump was operating, it maintained sufficient flow to ensure that all the specimens in a tank were immersed in sea water. When the timer shut the pump off, the tank was quickly emptied through a gravity drain. The sea water, which had a temperature of 10°C throughout the experiment, was obtained directly from the Strait of Juan de Fuca and used on a once-through basis.

Each surviving specimen was removed from the stressing frame after its scheduled length of exposure, rinsed in deionized water, dried and tension tested in an Instron 4204 tensile testing machine in accordance with ASTM Standard E8-89[25]. The apparent tensile strength was then calculated on the basis of the original cross-sectional area. Finally, five specimens of each surface treatment, which were not stressed or exposed to sea water, were broken in the tensile testing machine to determine the initial breaking strength.

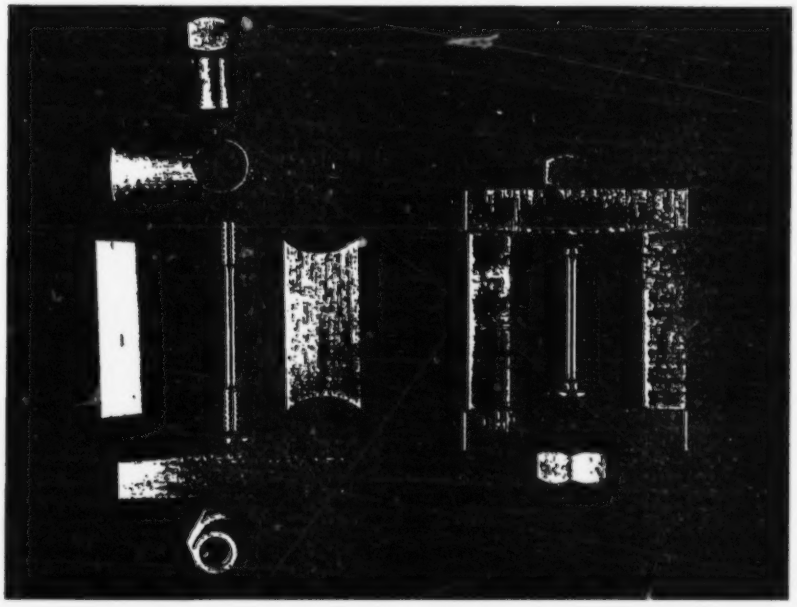

Figure 2. Exploded view of the components of the stressing frame, including the specimen, and the final stressed assembly.

## 5. RESULTS AND DISCUSSION

The residual breaking strengths of the uncoated, sulphuric acid anodized and hard anodized specimens exposed to alternate immersion in sea water are shown in Tables 2, 3 and 4, respectively. In these tables, a specimen that failed prior to tensile testing is marked by an "f" followed by the number of the day during which it failed or a "?" if that day was not determined. Ten uncoated specimens failed under the highest external load while no unstressed specimens failed, indicating that, as is already known, AA 7075-T651 is susceptible to stress corrosion cracking in sea water. Even without statistical analysis, the fact that only one hard anodized specimen failed suggests that hard anodizing improved the alloy's resistance to SCC. A total of five sulphuric acid anodized specimens failed, suggesting that sulphuric acid anodizing also had a beneficial effect, although not as pronounced as hard anodizing.

TABLE 2. Residual Breaking Strengths (MPa) of Uncoated AA 7075-T651 Specimens Exposed to Alternate Immersion in Sea Water

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa |
|---|---|---|---|---|
| Time (days) | | | | |
| 0 | 518<br>514<br>513<br>502<br>491 | | | |
| 2 | 507<br>507<br>502<br>499<br>496 | 516<br>507<br>502<br>500<br>497 | 510<br>507<br>503<br>481<br>472 | 472<br>469<br>452<br>451<br>329 |
| 4 | 511<br>506<br>499<br>498<br>498 | 513<br>506<br>504<br>502<br>489 | 521<br>497<br>496<br>473<br>396 | 511<br>476<br>453<br>449<br>431 |
| 7 | 500<br>487<br>487<br>486<br>480 | 456<br>411<br>212<br>173<br>150 | 504<br>382<br>285<br>f?<br>f? | f7<br>f7<br>f7<br>f?<br>f? |
| 10 | 536<br>464<br>457<br>419<br>398 | 388<br>252<br>197<br>175<br>58 | 245<br>234<br>f?<br>f?<br>f? | f?<br>f?<br>f?<br>f?<br>f? |

TABLE 3. Residual Breaking Strengths (MPa) of Sulphuric Acid Anodized AA 7075-T651 Specimens Exposed to Alternate Immersion in Sea Water

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa |
|---|---|---|---|---|
| Time (days) | | | | |
| 0 | 509<br>502<br>500<br>497<br>481 | | | |
| 2 | 513<br>506<br>501<br>501<br>499 | 512<br>509<br>508<br>499<br>489 | 508<br>496<br>493<br>490<br>464 | 505<br>491<br>428<br>417<br>353 |
| 4 | 507<br>505<br>504<br>498<br>494 | 525<br>499<br>494<br>492<br>484 | 504<br>503<br>502<br>375<br>358 | 420<br>417<br>400<br>361<br>270 |
| 7 | 512<br>507<br>506<br>498<br>487 | 503<br>487<br>480<br>473<br>462 | 493<br>478<br>266<br>189<br>180 | 505<br>427<br>354<br>342<br>214 |
| 10 | 538<br>520<br>505<br>501<br>486 | 498<br>404<br>219<br>167<br>156 | 196<br>124<br>115<br>93<br>f? | 222<br>f?<br>f?<br>f?<br>f? |

The calculated value of the 99% survival stress for each test group for each surface treatment is shown in Table 5, along with the correlation coefficient, r, of the best fit line determined for the plot of $E_i$ (Equation 4) versus the ranked breaking strengths. Specimens which were known to have failed during the last day of exposure were assigned a breaking strength equal to the externally-applied stress for these calculations. A sample calculation of $S_{99}$ appears in Appendix 1. A value of r=-1 indicates that the data within each cell followed the extreme value distribution exactly. Visual inspection of the r values reveals that this distribution did provide a reasonable description of the behaviour of the specimens within a test group. However, the sample size was too small to completely rule out other distributions.

The effects of the exposure time and the externally-applied stress on the values of $S_{99}$ are shown graphically in Figure 3 for

TABLE 4. Residual Breaking Strengths (MPa) of Hard Anodized AA 7075-T651 Specimens Exposed to Alternate Immersion in *Sea Water*

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa |
|---|---|---|---|---|
| Time (days) | | | | |
| 0 | 510<br>509<br>504<br>504<br>498 | | | |
| 2 | 515<br>511<br>507<br>504<br>503 | 522<br>511<br>510<br>507<br>505 | 519<br>505<br>504<br>503<br>472 | 514<br>511<br>504<br>502<br>436 |
| 4 | 518<br>515<br>506<br>502<br>499 | 511<br>509<br>507<br>505<br>499 | 514<br>508<br>499<br>434<br>434 | 436<br>426<br>413<br>314<br>224 |
| 7 | 520<br>511<br>511<br>507<br>505 | 514<br>512<br>504<br>502<br>500 | 516<br>509<br>507<br>503<br>498 | 514<br>501<br>277<br>274<br>f? |
| 10 | 514<br>506<br>499<br>499<br>495 | 507<br>504<br>504<br>489<br>460 | 522<br>422<br>332<br>237<br>210 | 479<br>327<br>237<br>219<br>207 |

uncoated specimens, Figure 4 for sulphuric acid anodized specimens and Figure 5 for hard anodized specimens. Comparison of these three figures does not reveal any substantial improvement in resistance to stress corrosion cracking as a result of either form of anodizing during the first four days of exposure to sea water. However, by the seventh day, an appreciable benefit from anodizing can be observed. Neither of the anodized groups showed any significant reduction in the value of $S_n$ for specimens exposed for 7 days at 69 MPa, while the value for the uncoated specimens had dropped to -632 MPa, which indicated that only the uncoated specimens were suffering from SCC at that stress level and length of exposure. By the tenth day, the sulphuric acid anodized specimens at 69 MPa had begun to suffer from SCC as well. The hard anodized specimens did not experience SCC at 69 MPa for any length of exposure.

TABLE 5. Calculated Values of 99% Survival Stress, $S_{99}$, and Correlation Coefficient, r, for Each Test Group for Each Surface Treatment

| | Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa |
|---|---|---|---|---|---|
| Time | | | | | |
| | | Uncoated AA 7075-T651 | | | |
| 0 days | $S_{99}$<br>r | 443<br>-0.979 | | | |
| 2 days | $S_{99}$<br>r | 475<br>-0.965 | 457<br>-0.916 | 393<br>-0.9532 | 51<br>-0.889 |
| 4 days | $S_{99}$<br>r | 463<br>-0.868 | 450<br>-0.969 | 190<br>-0.958 | 275<br>-0.928 |
| 7 days | $S_{99}$<br>r | 440<br>-0.879 | -632<br>-0.889 | -1260<br>-1.00 | -7790<br>- |
| 10 days | $S_{99}$<br>r | 131<br>-0.930 | -497<br>-0.964 | 48<br>-1.00 | -<br>- |
| | | Sulphuric Acid Anodized AA 7075-T651 | | | |
| 0 days | $S_{99}$<br>r | 436<br>-0.971 | | | |
| 2 days | $S_{99}$<br>r | 467<br>-0.876 | 449<br>-0.979 | 394<br>-0.969 | 80<br>-0.978 |
| 4 days | $S_{99}$<br>r | 470<br>-0.971 | 394<br>-0.866 | -33<br>-0.888 | 1<br>-0.957 |
| 7 days | $S_{99}$<br>r | 446<br>-0.986 | 391<br>-0.975 | -666<br>-0.888 | -257<br>-0.985 |
| 10 days | $S_{99}$<br>r | 394<br>-0.961 | -698<br>-0.887 | -339<br>-0.924 | -<br>- |
| | | Hard Anodized AA 7075-T651 | | | |
| 0 days | $S_{99}$<br>r | 478<br>-0.972 | | | |
| 2 days | $S_{99}$<br>r | 477<br>-0.921 | 468<br>-0.869 | 394<br>-0.937 | 286<br>-0.890 |
| 4 days | $S_{99}$<br>r | 459<br>-0.938 | 481<br>-0.990 | 224<br>-0.900 | -180<br>-0.962 |
| 7 days | $S_{99}$<br>r | 474<br>-0.896 | 468<br>-0.922 | 467<br>-0.976 | -1070<br>-0.894 |
| 10 days | $S_{99}$<br>r | 456<br>-0.907 | 373<br>-0.941 | -438<br>-0.946 | -475<br>-0.843 |

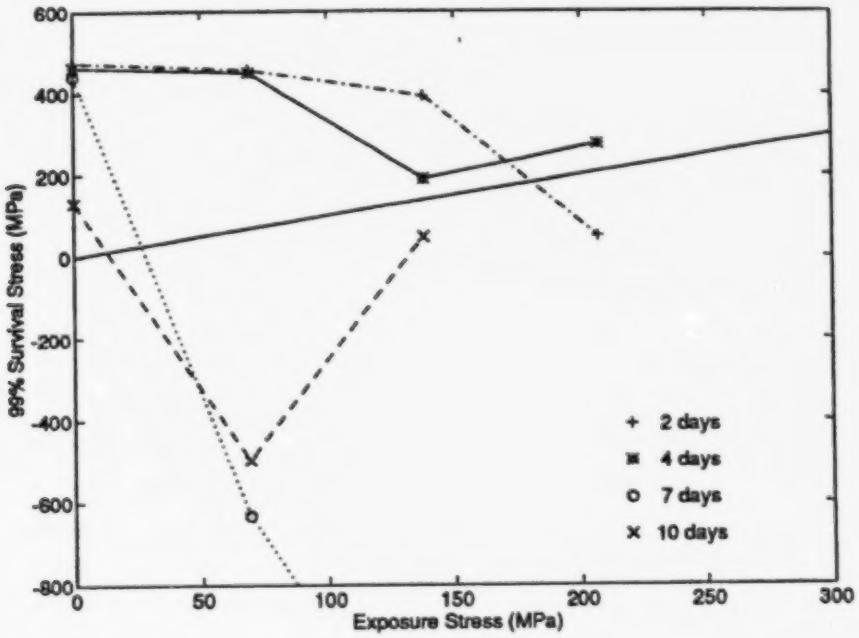


Figure 3. Plot of 99% survival stress versus externally-applied exposure stress for uncoated specimens of AA 7075-T651.

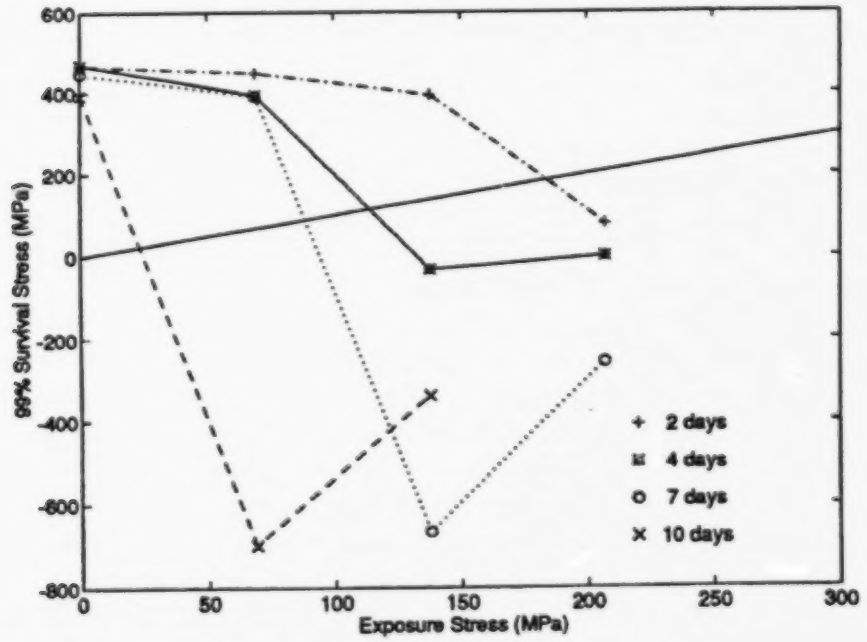


Figure 4. Plot of 99% survival stress versus externally-applied exposure stress of sulphuric acid anodized specimens of AA 7075-T651.

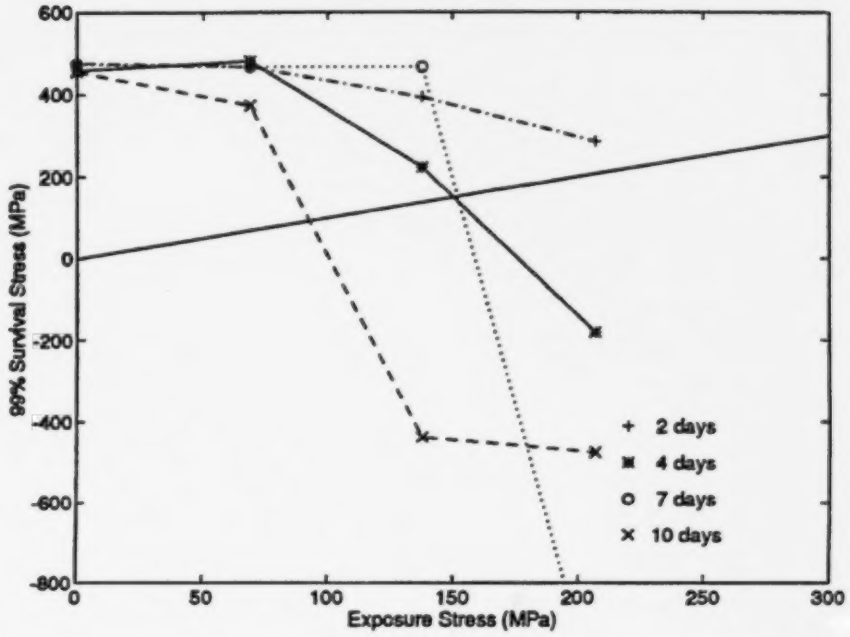


Figure 5. Plot of 99% survival stress versus externally-applied exposure stress of hard anodized specimens of AA 7075-T651.

Any conclusions about the extent of SCC at higher stress levels are clouded by artifacts generated as a result of the method used to treat failed specimens in the statistical procedure. These artifacts include the marked increase in the value of $S_{99}$ for the uncoated specimens exposed for 10 days at 138 MPa compared with those at the lower stress level of 69 MPa.

Figure 3 shows a substantial reduction in the value of $S_{99}$ for the uncoated specimens exposed to sea water for 10 days in the absence of an externally-applied stress (0 MPa), both in comparison with the three shorter exposure times and with the equivalent 10 day exposure for the anodized specimens. This reduction is suggestive of the onset of intergranular cracking, which could in fact be observed using metallographic techniques (Figure 6). Thus both sulphuric acid and hard anodizing at a minimum delayed the onset of intergranular cracking.

Depending on the choice of which values of $S_{99}$ should be used in the calculation of the statistically-significant threshold stress, either a pessimistic or an optimistic value could be obtained. A sample calculation appears in Appendix 2. The results are summarized in Table 6. The values of $S_{th}$ for the uncoated specimens are well within

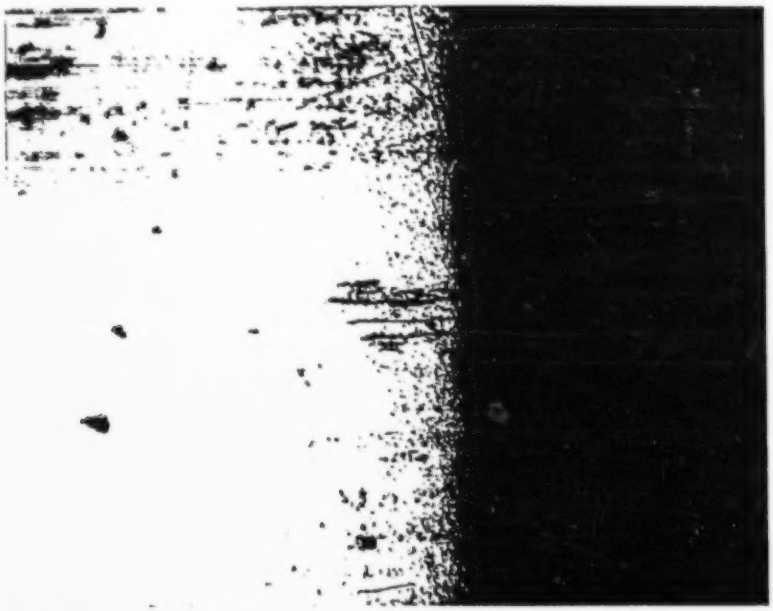

Figure 6. Intergranular cracking of uncoated AA 7075-T651 exposed to alternate immersion in sea water for 10 days. Etched with Keller's reagent. 250x.

the range reported by the interlaboratory test program[11], which varied from -119.5 MPa to 59.4 MPa. The ranges of the values for the different surface treatments do not overlap and lead to the conclusion that both sulphuric acid and hard anodizing improve the resistance of AA 7075-T651 to stress corrosion cracking, with hard anodizing providing the greater benefit.

As noted in the introduction to the Box-Cox transformation, successful implementation of the transformation is based on the assumption that the natural logarithm of the standard deviation of each individual stress/time test group increases linearly with decreasing

TABLE 6. Threshold Stresses for Each of the Three Surface Treatments

| Surface Treatment | Threshold Stress (MPa) | |
|---|---|---|
| | Pessimistic | Optimistic |
| Uncoated | -47 | -13 |
| Sulphuric Acid Anodized | 15 | 23 |
| Hard Anodized | 52 | 71 |

TABLE 7. Slope of ln (Standard Deviation) versus ln (Mean of Normalized Breaking Strengths), Correlation Coefficient and Power Parameter for Each of the Three Surface Treatments

| Surface Treatment | Slope ($\alpha$) | Correlation Coefficient (r) | Power Parameter ($\lambda$) |
|---|---|---|---|
| Uncoated | -2.02 | -0.487 | 3.02 |
| Sulphuric Acid Anodized | -1.78 | -0.546 | 2.78 |
| Hard Anodized | -6.99 | -0.873 | 7.99 |

natural logarithm of the mean breaking strength. The correlation coefficients shown in Table 7 indicate a rather poor fit to the data for the uncoated and sulphuric acid anodized specimens. Nevertheless, the slopes did result in power parameters that served to stretch the residual strength axis for large strength values and compress the axis for small values. However, the transformed data may not follow a normal distribution or have variances for the individual stress/time groups that are approximately equal. As a result, calculations of the least significant difference and the analysis of variance for the uncoated and sulphuric acid anodized specimens may not be strictly valid.

The mean of the Box-Cox transformed data for each stress/time group for the uncoated specimens is plotted in Figure 7 as a function of the externally-applied stress. Similar graphs for the sulphuric acid anodized and hard anodized specimens appear in Figure 8 and Figure 9, respectively. Each graph also shows the value of the least significant difference, LSD, at the 95% confidence level for that surface treatment. These values were found to be 25.4 for the uncoated specimens, 28.2 for the sulphuric acid anodized specimens and 34.4 for the hard anodized specimens.

The Box-Cox transformation served to confirm several of the conclusions that were reached using the original breaking load method and removed the anomalous behaviour that was observed at higher stress levels as a result of the original statistical procedure. As suggested by the original method, a statistically-significant reduction in the breaking strength at the 69 MPa stress level occurred by the $7^{th}$ day of exposure for the uncoated specimens, occurred between the $7^{th}$ and $10^{th}$

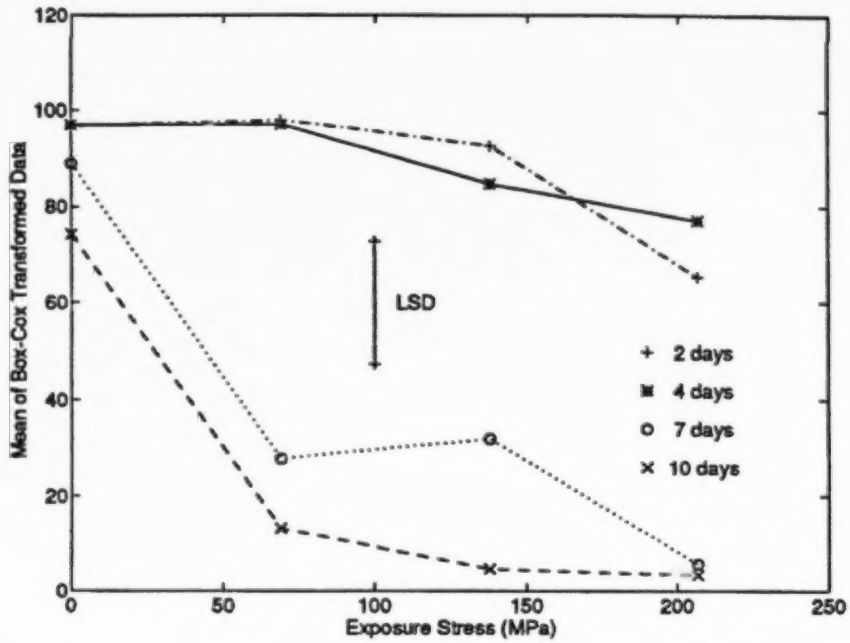


Figure 7. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for uncoated AA 7075-T651.

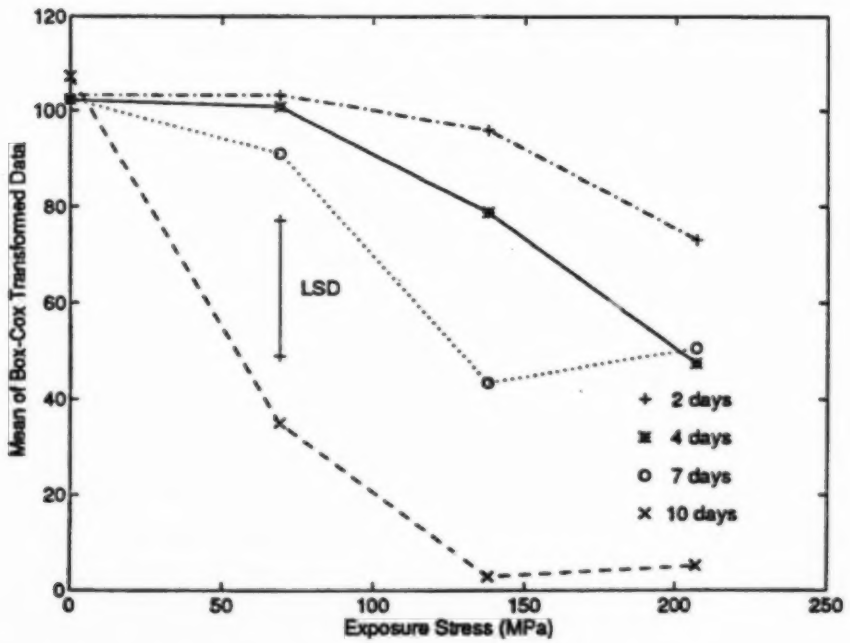


Figure 8. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for sulphuric acid anodized AA 7075-T651.

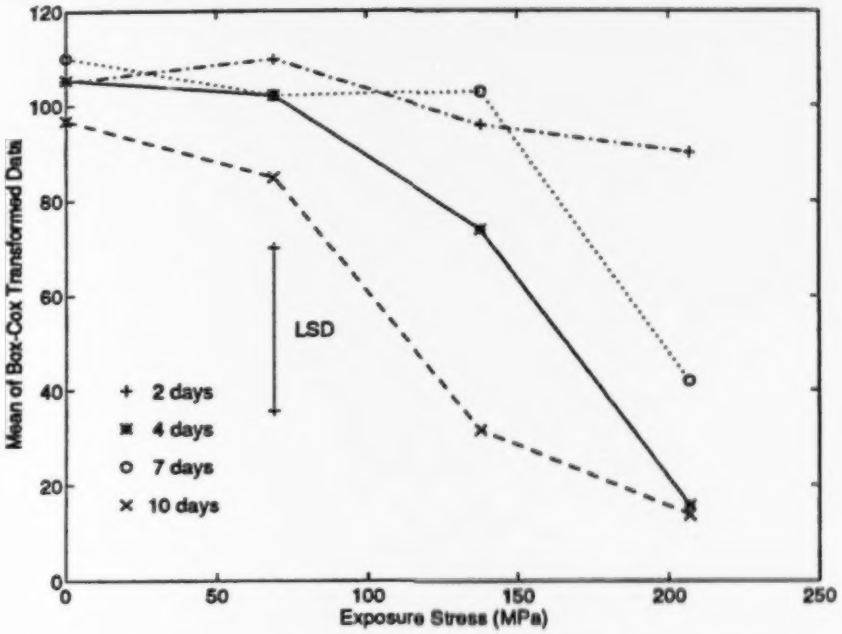


Figure 9. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for hard anodized AA 7070-T651.

days for the sulphuric acid anodized specimens and did not occur at all for the hard anodized specimens. At the 138 MPa stress level, the Box-Cox transformation showed a significant reduction in the breaking strength by the 7$^{th}$ day for the uncoated specimens, by the 7$^{th}$ day for the sulphuric acid anodized specimens as well, and between the 7$^{th}$ and 10$^{th}$ days for the hard anodized specimens. The hard anodizing therefore continued to offer improved resistance to SCC at this higher stress level. At the 207 MPa stress level, the transformation showed a significant reduction in the breaking strength of the uncoated and sulphuric acid anodized specimens within two days of exposure that was not observed for the hard anodized specimens, again demonstrating the improvement provided by hard anodizing.

The behaviour of the 4 day, 207 MPa test group of uncoated specimens was assumed to be anomalous given the significant reduction in breaking strength already observed after 2 days at that stress level. Such results will occasionally occur as a result of the small sample size within each test group.

The difference between the means of the transformed data for

the 10 day, 0 Mpa group and the control group (0 days, 0 MPa) for the uncoated specimens was found to be 25.9, which was slightly greater than the LSD of 25.4, and indicated that the reduction in breaking strength was statistically significant. As shown in Figure 6, this reduction was caused by the onset of intergranular cracking. Any reduction in the breaking strength of the sulphuric acid or hard anodized specimens at 0 MPa was not significant and intergranular cracking was not detected on these specimens.

An analysis of variance table involving all three variables (stress level, exposure time and surface treatment) for the transformed data appears in Table 8. In this table, the four means appearing under the four different applied stress levels were calculated using all specimens that were tested under each load, independent of the length of exposure and the surface treatment. Similarly, the four means appearing under the four different exposure times involved all specimens for each exposure time, independent of the applied load and the surface treatment. Finally, the three means appearing under the three different surface treatments involved all specimens for each surface treatment, independent of the stress level and length of exposure. The error shown under the grand (overall) mean represents the standard error for each stress/time test group for each alloy. The standard error for each treatment mean appears in brackets beside that mean. The table shows that the addition of an externally-applied stress had a significant effect on the degradation of AA 7075-T651 specimens exposed to alternate immersion in sea water. Furthermore, the degradation increased with increasing level of stress. The table also shows that the degradation increased with each increasing length of exposure. Finally, anodizing is shown to provide a significant improvement in resistance to SCC, with hard anodizing providing a greater benefit than sulphuric acid anodizing.

Unfortunately, an overall table like Table 8 masks some of the differences in behaviour resulting from the different surface treatments. For example, the table indicates that an external load of 69 MPa resulted in a significant decrease in breaking strength. However, an ANOVA table for the hard anodized specimens by themselves shows that a load of 69 MPa did not significantly affect the breaking strength of these specimens (Appendix 3a). Significant SCC did not occur until the 138 MPa stress level. The extent of damage at the 69 MPa level on the uncoated specimens was so severe (Appendix 3b) that it caused the difference in the treatment means between 0 MPa and 69 MPa to be significant when all three treatments were combined in Table 8.

Table 8. Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths for All Three Surface Treatments

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa | Grand Mean |
|---|---|---|---|---|---|
| Mean | 99.2 (3.0) | 80.5 (3.0) | 61.6 (3.0) | 40.8 (3.0) | 70.5 |
| Time | 2 days | 4 days | 7 days | 10 days | Error |
| Mean | 94.1 (3.0) | 81.9 (3.0) | 66.6 (3.0) | 39.4 (3.0) | 10.3 |
| Surface Treatment | Uncoated | Sulphuric Acid Anodized | Hard Anodized | | |
| Mean | 59.9 (2.6) | 71.5 (2.6) | 80.2 (2.6) | | |
| Source of Variation | Degrees of Freedom | Sum of Squares | Mean Square | F ratio | $F_{0.95}$ |
| Mean | 1 | 1190000 | 1190000 | | |
| Stress | 3 | 113000 | 37600 | 71.2 | 3.95 |
| Time | 3 | 100000 | 33400 | 63.2 | 3.95 |
| Surface Treatment | 2 | 16600 | 8280 | 15.6 | 4.79 |
| Stress-Time Interaction | 9 | 28400 | 3150 | 6.0 | 2.56 |
| Stress-Surface Interaction | 6 | 9860 | 1640 | 3.1 | 2.96 |
| Time-Surface Interaction | 6 | 2460 | 4100 | 7.8 | 2.96 |
| Three Factor Interaction | 18 | 17900 | 994 | 1.9 | 2.19 |
| Error | 192 | 102000 | 529 | | |
| Total | 240 | 1610000 | | | |

## 6. CONCLUSIONS

The breaking load method showed that, at a minimum, anodizing delayed the onset of stress corrosion cracking for specimens exposed to alternate immersion in sea water at 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an externally-applied stress. Intergranular cracking was observed on the uncoated specimens after 10 days of exposure but not on any of the anodized specimens. The method also yielded values for a statistically valid threshold stress for each surface treatment that

supported the conclusion that both sulphuric acid and hard anodizing improve the resistance of AA 7075-T651 to SCC, with hard anodizing providing the greater benefit. The value of $S_{th}$ for the uncoated specimens was well within the range reported by an interlaboratory program designed to test the reproducibility of the method and to evaluate alternative statistical procedures.

The extreme value distribution was found to provide a reasonably good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory test program. The procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusions reached using the original method, even though the statistical calculations may not have been strictly valid. It also showed that hard anodizing continued to offer improved resistance to SCC at higher stress levels.

Analysis of variance of the transformed data confirmed the conclusion that anodizing provided a significant improvement in resistance to SCC, with hard anodizing providing the greater benefit. Thus, the decision to hard anodize the aluminum in the pressure hull of the underwater vehicle has been supported by these experiments. Furthermore, the hard anodizing provides an excellent substrate for a paint system which would result in an even greater reduction in the possibility of unexpected failure due to stress corrosion cracking.

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APPENDIX 1. Sample Calculation of 99% Survival Stress

The ranked breaking strengths for the five uncoated specimens exposed to sea water for 4 days at 138 MPa are shown in the following table, along with the expected value of each ranked observation, $E_i$.

| Observation i | $R_i = (i-\frac{1}{2})/5$ | $E_i = -\ln[-\ln(R_i)]$ | Breaking Strength (MPa) |
|---|---|---|---|
| 1 | 0.1 | -0.834 | 521 |
| 2 | 0.3 | -0.186 | 497 |
| 3 | 0.5 | 0.367 | 496 |
| 4 | 0.7 | 1.031 | 473 |
| 5 | 0.9 | 2.250 | 396 |

A plot of $E_i$ versus the ranked breaking strengths yields a best-fit line with slope, m, equal to -0.0235, intercept, b, equal to 11.7, and correlation coefficient, r, equal to -0.958. The value of r near -1 indicates that the extreme value distribution provides a good fit to the data.

Initial estimates of the distribution location and scale parameters, $\beta$ and $\delta$, are found to be:

$$\beta_o = -\frac{b}{m} = 499 \tag{1}$$

$$\delta_o = -\frac{1}{m} = 42.5 \tag{2}$$

These parameters are adjusted to account for the small sample size using the following equations:

$$\delta = \delta_o(\sigma_o/\sigma_n) = 42.5((\pi/\sqrt{6})/0.7932) = 68.7 \tag{3}$$

$$\begin{aligned}\beta &= \beta_o - \delta_o y_o + \delta \bar{y}_n \\ &= 499 - 42.5(0.577) + 68.7(0.4565) \\ &= 506\end{aligned} \tag{4}$$

The value of the 99% survival stress, $S_{99}$, is then calculated:

$$\begin{aligned}S_{99} &= \beta - 4.60 \cdot \delta \\ &= 506 - 4.60\ (68.7) \\ &= 190\ MPa\end{aligned} \tag{5}$$

APPENDIX 2. Sample Calculation of the Threshold Stress, $S_{th}$.

The first step in determining the statistically-defined threshold stress involves calculation of the slope, B, and intercept, A, of the best-fit line obtained from a plot of the 99% survival stress, $S_{99}$, versus the exposure stress, $S_{exp}$, for each length of exposure. The point of intersection of this line with the line defined by $S_{99} = S_{exp}$ is given by:

$$S'_{th} = -\frac{A}{B-1} \qquad (1)$$

As noted in the discussion, a range of values can be obtained depending on whether or not anomalous points arising from the statistical procedure are included in the calculation. Inclusion of these points results in a higher value of $S_{th}$ than would be obtained without them. The results for the sulphuric acid anodized specimens with these anomalous points included appear in the following table:

| Exposure Length | Slope | Intercept | $S'_{th}$ (MPa) |
|---|---|---|---|
| 2 days | -1.76 | 530 | 192 |
| 4 days | -2.66 | 483 | 132 |
| 7 days | -4.59 | 454 | 81.1 |
| 10 days | -5.31 | 152 | 24.1 |

The mean, m, and standard deviation, s, of the values of $S'_{th}$ are determined and used to calculate $S_{th}$:

$$\begin{aligned} S_{th} &= m - t_{0.95(3)} \cdot \frac{s}{\sqrt{4}} \\ &= 107 - 2.353 \cdot \frac{71}{\sqrt{4}} \\ &= 23\ MPa \end{aligned} \qquad (2)$$

If the 4 day, 207 MPa point, the 7 day, 207 MPa point and the 10 day, 138 MPa point are removed from the calculation, the results become:

| Exposure Length | Slope | Intercept | $S'_{th}$ (MPa) |
|---|---|---|---|
| 2 days | -1.76 | 530 | 192 |
| 4 days | -3.64 | 528 | 114 |
| 7 days | -8.07 | 613 | 67.7 |
| 10 days | -15.8 | 394 | 23.4 |

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APPENDIX 2. (cont.)

The value of $S_{th}$ was then found to be:

$$S_{th} = 99 - 2.353 \cdot \frac{72}{\sqrt{4}} \qquad (3)$$
$$= 15 \ MPa$$

APPENDIX 3(a). Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths of Hard Anodized Specimens of AA 7075-T651.

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa | Grand mean: |
|---|---|---|---|---|---|
| Mean | 104 (6.2) | 99.8 (6.2) | 76.1 (6.2) | 40.4 (6.2) | 80.2 |
| Time | 2 days | 4 days | 7 days | 10 days | Error |
| Mean | 100 (6.2) | 74.4 (6.2) | 89.3 (6.2) | 56.8 (6.2) | 12.4 |
| Source of Variation | Degrees of Freedom | Sum of Squares | Mean Square | F ratio | $F_{0.99}$ |
| Mean | 1 | 514000 | 514000 | | |
| Stress | 3 | 51300 | 17100 | 22.2 | 4.13 |
| Time | 3 | 21300 | 7110 | 9.21 | 4.13 |
| Interaction | 9 | 15200 | 1690 | 2.19 | 2.72 |
| Error | 64 | 49400 | 772 | | |
| Total | 80 | 652000 | | | |

APPENDIX 3(b). Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths of Uncoated Specimens of AA 7075-T651.

| Stress | 0 MPa | 69 MPa | 138 MPa | 207 MPa | Grand Mean |
|---|---|---|---|---|---|
| Mean | 89.2 (4.1) | 59.1 (4.1) | 53.5 (4.1) | 37.9 (4.1) | 59.9 |
| Time | 2 days | 4 days | 7 days | 10 days | Error |
| Mean | 88.2 (4.1) | 89.0 (4.1) | 38.5 (4.1) | 23.9 (4.1) | 8.1 |
| Source of Variation | Degrees of Freedom | Sum of Squares | Mean Square | F ratio | $F_{0.99}$ |
| Mean | 1 | 287000 | 287000 | | |
| Stress | 3 | 27700 | 9240 | 28.2 | 4.13 |
| Time | 3 | 68100 | 22700 | 69.1 | 4.13 |
| Interaction | 9 | 13400 | 1480 | 4.52 | 2.72 |
| Error | 64 | 21000 | 328 | | |
| Total | 80 | 417000 | | | |

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13. ABSTRACT ( a brief and factual summary of the document. It may also appear elsewhere in the body of the document itself. It is highly desirable that the abstract of classified documents be unclassified. Each paragraph of the abstract shall begin with an indication of the security classification of the information in the paragraph (unless the document itself is unclassified) represented as (S), (C), or (U). It is not necessary to include here abstracts in both official languages unless the text is bilingual).

The breaking load method was used to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens of AA 7075-T651 in order to evaluate the benefits of anodizing the aluminum pressure hull of an autonomous underwater vehicle to reduce the risk of stress corrosion cracking. The original test method showed that anodizing delayed the onset of stress corrosion cracking of specimens subjected to an externally-applied stress of 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an external stress. The value of the threshold stress determined for the uncoated specimens was within the range reported by an interlaboratory test program.

The extreme value distribution was found to provide a good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory program. A new statistical procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusion reached using the original method, even though the statistical calculations may not have been strictly valid. The new procedure also showed that hard anodizing continued to offer improved resistance to stress corrosion cracking at higher stress levels. Finally, analysis of variance of the transformed data confirmed the conclusion that anodizing provided a significant improvement in resistance to stress corrosion cracking, with hard anodizing providing the greater benefit.

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stress corrosion cracking

aluminum

sea water

breaking load

anodizing

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